

**COMMERCIAL-SCALE DEMONSTRATION OF THE
LIQUID PHASE METHANOL (LPMEOH™) PROCESS**

TECHNICAL PROGRESS REPORT NO. 28

For The Period

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and

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for the

Air Products Liquid Phase Conversion Company, L.P.

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Abstract

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was built at a site located at the Eastman chemicals-from-coal complex in Kingsport.

The LPMEOH™ Demonstration Unit operated at 100% availability during this quarter. There were four syngas outages experienced during the quarter. The planned biennial LPMEOH™ Demonstration Unit outage, which began on 28 March 2001, was completed during this quarter on 20 April 2001. The LPMEOH™ Demonstration Unit was also shutdown for the planned biennial Eastman complex-wide outage on 13 May 2001 and restarted on 22 May 2001.

The scheduled biennial inspection of all pressure vessels within the LPMEOH™ Demonstration Unit, as required by Tennessee state code, was performed. Catalyst slurry was pressure transferred from the LPMEOH™ Reactor to the slurry tank (29D-02) and held during the outage period. No issues were observed with any of the units evaluated as part of the code inspection. Maintenance activities associated with instrumentation and a sample point to the gas chromatographs were performed during this outage. The engineering modifications required for the reduction of the adsorbent in the 29C-40 catalyst guard bed and in-situ activation of methanol synthesis catalyst in the 29C-01 LPMEOH™ Reactor were also completed during the shutdown period.

The LPMEOH™ Demonstration Unit was also shut down for the biennial Eastman complex-wide outage on 13 May 2001. During this outage, the catalyst slurry was held in the LPMEOH™ Reactor and the recycle compressor was used to maintain reactor inlet flow as part of the standby conditions. No maintenance activities were conducted within the LPMEOH™ Demonstration Unit during this outage.

A catalyst withdrawal and addition campaign was undertaken during the quarter to raise catalyst activity. A series of two withdrawals were conducted on 04 and 05 June 2001. Two batches of fresh catalyst were activated and added to the reactor on 07 and 08 June 2001, respectively. After the second batch of freshly activated catalyst was transferred into the reactor, the catalyst inventory was calculated to be 45,620 pounds. There were no operating periods of sufficient duration (minimum of about 2 weeks) to calculate a catalyst deactivation rate.

During the reporting period, quantities of the syngas stream which contains primarily carbon monoxide (Carbon Monoxide Gas, or CO Gas) became available for use in CO-rich testing. A reactor inlet ratio of hydrogen (H₂) to CO of approximately 0.6:1 was

used for operations conducted between 09 May 2001 and 13 May 2001. The methanol productivity met expectations for this condition.

During most of the quarter, the flowrate of the primary syngas feed (Balanced Gas) was controlled at an average flowrate of 575 KSCFH. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235°C.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Copper crystallite size measurements have not increased in the most recent samples (an increase may be an indication of catalyst deactivation). Additional updates to the catalyst poisons data are pending further analytical results.

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. During the biennial outage, the sparger was removed from the LPMEOH™ Reactor for inspection and cleaning. In addition, all differential pressure transmitters around the sparger and reactor were calibrated, and the pressure taps were cleaned. Following the restart of the unit, the resistance coefficient was higher than the value which was calculated following the March 1999 outage. The performance of the sparger will continue to be monitored closely for changes in performance.

During this reporting period, the adsorbent material in the 29C-40 catalyst guard bed was brought onstream. This material is chemically treated with copper oxide to enhance its arsine removal capabilities. Earlier work had identified the need to reduce the copper oxide component to copper metal prior to exposure to syngas at full header pressure. The reduction procedure, using a dilute stream of Balanced Gas in nitrogen and a protocol for increasing the adsorbent temperature with time, was begun on 17 April 2001. Temperature control within the catalyst guard bed was excellent. The reduction ended on 19 April 2001. The catalyst guard bed was then cooled, and a pressure check to full header pressure with syngas was conducted. The guard bed internal temperatures increased by about 30-40°C and then stabilized following the introduction of the syngas. This was consistent with the temperature rise which would be expected due to the adsorption of CO on the guard bed material.

The catalyst guard bed was placed in service at 1500 hrs on 26 April 2001. There were no incidents resulting from pressurizing or initial contact with the Balanced Gas feed stream. During the week of 30 April 2001, Air Products analytical personnel monitored the initial performance of the catalyst guard bed for removal of arsenic. Preliminary results indicated that breakthrough of arsine may have already occurred. Additional laboratory and field test work is ongoing to confirm these results as well as determine the mechanism for the early breakthrough of arsine through the catalyst guard bed. It is possible that damage to the adsorbent material may have occurred during the August 2000 temperature excursion when this material was first placed into service. Pending results of the additional testing, a decision to remove and replace the existing adsorbent will be made.

During the reporting period, a total of 2,655,172 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 79.1 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations were concluded at the project sites selected for the off-site, product-use test program. Work has begun on the draft Topical Report which summarizes the objectives and results of this product-use test program.

Activities associated with Design Verification Testing (DVT) of the Liquid Phase Dimethyl Ether (LPDME) Process have been completed. A Topical Report, which presents the results of the DVT at the LaPorte AFDU, has been issued. The LPMEOH™ Demonstration Project will prepare a separate Topical Report on the market analysis for DME and review of the economics of the LPDME Process.

A DOE quarterly review meeting was held during the week of 25 June 2001 in Pittsburgh. The performance of the LPMEOH™ Demonstration Unit since the last meeting (January 2001) was the primary topic of discussion.

Air Products has been asked to participate in four conferences where the results of the LPMEOH™ Demonstration Project will be presented. These meetings include the U.S.-China Clean Energy Technology Forum (29 August - 01 September 2001), the Gasification Technologies Conference (07-10 October 2001), the Clean Coal and Power Conference (19-20 November 2001), and the 18th Annual International Pittsburgh Coal Conference (03-07 December 2001). All abstracts have been approved by DOE, and the papers are at various stages of development.

One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2001. Seventy-five percent (75%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2001.

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ACRONYMS AND DEFINITIONS

Acurex	-	Acurex Environmental Corporation (now ARCADIS Geraghty & Miller)
Air Products	-	Air Products and Chemicals, Inc.
AFDU	-	Alternative Fuels Development Unit - The "LaPorte PDU"
AFFTU	-	Alternative Fuels Field Trailer Unit
Balanced Gas	-	A syngas with a composition of hydrogen (H ₂), carbon monoxide (CO), and carbon dioxide (CO ₂) in stoichiometric balance for the production of methanol
Btu	-	British Thermal Unit
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
Catalyst Activity	-	the rate at which the catalyst promotes the desired chemical reaction to proceed within the limitations of chemical equilibrium
Catalyst Age (η -eta)	-	the ratio of the rate constant at any point in time to the rate constant for a freshly reduced catalyst (as determined in the laboratory autoclave)
Catalyst Concentration	-	Synonym for Slurry Concentration
Catalyst Loading	-	Synonym for Slurry Concentration
CO Conversion	-	the percentage of CO consumed across the reactor
Crude Grade Methanol	-	Underflow from rectifier column (29C-20), defined as 80 wt% minimum purity; requires further distillation in existing Eastman equipment prior to use
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DOE-NETL	-	The DOE's National Energy Technology Laboratory (Project Team)
DOE-HQ	-	The DOE's Headquarters - Coal Fuels and Industrial Systems (Project Team)
DTP	-	Demonstration Test Plan - The Operating Plan for Phase 3, Task 2 Operation
DVT	-	Design Verification Testing
Eastman	-	Eastman Chemical Company
EIV	-	Environmental Information Volume
EMP	-	Environmental Monitoring Plan
EPRI	-	Electric Power Research Institute
FFV	-	flexible-fuel vehicle
Fresh Feed	-	sum of Balanced Gas, H ₂ Gas, and CO Gas
Gas Holdup	-	the percentage of reactor volume up to the Gassed Slurry Height which is gas
Gassed Slurry Height	-	height of gassed slurry in the reactor
HAPs	-	Hazardous Air Pollutants
Hydrogen Gas	-	A syngas containing an excess of hydrogen (H ₂) over the stoichiometric balance for the production of methanol; also called H ₂ Gas
IGCC	-	Integrated Gasification Combined Cycle, a type of electric power generation plant
IGCC/OTM	-	An IGCC plant with a "Once-Thru Methanol" plant (the LPMEOH™ Process) added-on
Inlet Superficial Velocity	-	the ratio of the actual cubic feet of gas at the reactor inlet (calculated at the reactor temperature and pressure) to the reactor cross-sectional area (excluding the area contribution by the internal heat exchanger); typical units are feet per second
K	-	Sparger resistance coefficient (term used in calculation of pressure drop)
KSCFH	-	Thousand Standard Cubic Feet per Hour
LaPorte PDU	-	The DOE-owned experimental unit (PDU) located adjacent to Air Products' industrial gas facility at LaPorte, Texas, where the LPMEOH™ Process was successfully piloted
LPDME	-	Liquid Phase DME Process, for the production of DME as a mixed coproduct with methanol
LPMEOH™	-	Liquid Phase Methanol (the technology to be demonstrated)
M85	-	a fuel blend of 85 volume percent methanol and 15 volume percent unleaded gasoline
MeOH	-	methanol
Methanol Productivity	-	the gram-moles of methanol produced per hour per kilogram catalyst (on an oxide basis)
MW	-	molecular weight, pound per pound mole

ACRONYMS AND DEFINITIONS (cont'd)

NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration
ρ	-	density, pounds per cubic foot
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit
PFD	-	Process Flow Diagram(s)
ppbv	-	parts per billion (volume basis)
ppmw	-	parts per million (weight basis)
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psi	-	pounds per square inch
psia	-	pounds per square inch (absolute)
psig	-	pounds per square inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
Raw Methanol	-	sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol which is produced after stabilization
Reactor Feed	-	sum of Fresh Feed and Recycle Gas
Reactor O-T-M Conversion	-	percentage of energy (on a lower heating value basis) in the Reactor Feed converted to methanol (Once-Through-Methanol basis)
Reactor Volumetric Productivity	-	the quantity of Raw Methanol produced (tons per day) per cubic foot of reactor volume up to the Gassed Slurry Level
Recycle Gas	-	the portion of unreacted syngas effluent from the reactor “recycled” as a feed gas
Refined Grade Methanol	-	Distilled methanol, defined as 99.8 wt% minimum purity; used directly in downstream Eastman processes
SCF	-	Standard Cubic Feet
SCFH	-	Standard Cubic Feet per Hour
Slurry Concentration	-	percentage of weight of slurry (solid plus liquid) which is catalyst (on an oxide basis)
Sl/hr-kg	-	Standard Liter(s) per Hour per Kilogram of Catalyst
Syngas	-	Abbreviation for Synthesis Gas
Syngas Utilization	-	defined as the number of standard cubic feet of Balanced Gas plus CO Gas to the LPMEOH™ Demonstration Unit required to produce one pound of Raw Methanol
Synthesis Gas	-	A gas containing primarily hydrogen (H ₂) and carbon monoxide (CO), or mixtures of H ₂ and CO; intended for "synthesis" in a reactor to form methanol and/or other hydrocarbons (synthesis gas may also contain CO ₂ , water, and other gases)
Tie-in(s)	-	the interconnection(s) between the LPMEOH™ Process Demonstration Unit and the Eastman Facility
TPD	-	Ton(s) per Day
V	-	volumetric flowrate, thousand standard cubic feet per hour
VOC	-	volatile organic compound
vol%	-	volume %
WBS	-	Work Breakdown Structure
wt	-	weight

Executive Summary

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was designed, constructed, and is in operation at a site located at the Eastman chemicals-from-coal complex in Kingsport.

On 04 October 1994, Air Products and Eastman signed the agreements that would form the Partnership, secure the demonstration site, and provide the financial commitment and overall project management for the project. These partnership agreements became effective on 15 March 1995, when DOE authorized the commencement of Budget Period No. 2 (Modification No. A008 to the Cooperative Agreement). The Partnership has subcontracted with Air Products to provide the overall management of the project, and to act as the primary interface with DOE. As subcontractor to the Partnership, Air Products provided the engineering design, procurement, construction, and commissioning of the LPMEOH™ Process Demonstration Unit, and is providing the technical and engineering supervision needed to conduct the operational testing program required as part of the project. As subcontractor to Air Products, Eastman is responsible for operation of the LPMEOH™ Process Demonstration Unit, and for the interconnection and supply of syngas, utilities, product storage, and other needed services.

The project involves the operation of an 80,000 gallons per day (260 tons per day (TPD)) methanol unit utilizing coal-derived syngas from Eastman's integrated coal gasification facility. The new equipment consists of syngas feed preparation and compression facilities, the liquid phase reactor and auxiliaries, product distillation facilities, and utilities.

The technology to be demonstrated is the product of a cooperative development effort by Air Products and DOE in a program that started in 1981. Developed to enhance electric power generation using integrated gasification combined cycle (IGCC) technology, the LPMEOH™ Process is ideally suited for directly processing gases produced by modern day coal gasifiers. Originally tested at the Alternative Fuels Development Unit (AFDU), a small, DOE-owned experimental unit in LaPorte, Texas, the technology provides several improvements essential for the economic coproduction of methanol and electricity directly from gasified coal. This liquid phase process suspends fine catalyst particles in an inert liquid, forming a slurry. The slurry dissipates the heat of the chemical reaction away from the catalyst surface, protecting the catalyst and allowing the methanol synthesis reaction to proceed at higher rates.

At the Eastman chemicals-from-coal complex, the technology is integrated with existing coal gasifiers. A carefully developed test plan will allow operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable

on/off operation, and its ability to produce methanol as a clean liquid fuel without additional upgrading. An off-site, product-use test program was conducted to demonstrate the suitability of the methanol product as a transportation fuel and as a fuel for stationary applications for small modular electric power generators for distributed power.

The operating test phase and the completed off-site product-use test program have been developed to demonstrate the commercial viability of the LPMEOH™ Process and allow utilities to evaluate the application of this technology in the coproduction of methanol with electricity. A typical commercial-scale IGCC coproduction facility, for example, could be expected to generate 200 to 350 MW of electricity, and to also manufacture 45,000 to 300,000 gallons per day of methanol (150 to 1,000 TPD). A successful demonstration at Kingsport will show the ability of a local resource (coal) to be converted in a reliable (storable) and environmentally preferable way to provide the clean energy needs of local communities for electric power and transportation.

This project has also completed design verification testing (DVT), including laboratory- and pilot-scale research and market verification studies, to evaluate whether to include a demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol. DME has several commercial uses. In a storable blend with methanol, the mixture can be used as a peaking fuel in gasification-based electric power generating facilities, or as a diesel engine fuel. Blends of methanol and DME can be used as chemical feedstocks for synthesizing chemicals, including new oxygenated fuel additives.

The project was reinitiated in October of 1993, when DOE approved a site change to the Kingsport location. DOE conditionally approved the Continuation Application to Budget Period No. 2 (Design and Construction) in March of 1995 and formally approved it on 01 June 1995 (Modification No. M009). After approval, the project initiated Phase 1 - Design - activities. Phase 2 - Construction - activities were initiated in October of 1995. The project required review under the National Environmental Policy Act (NEPA) to move to the construction phase. DOE prepared an Environmental Assessment (DOE/EA-1029), and subsequently a Finding of No Significant Impact (FONSI) was issued on 30 June 1995. The Cooperative Agreement was modified (Modification No. A011) on 08 October 1996, authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). This modification provides the full \$213,700,000 of authorized funding, with 56.7% participant cost share and 43.3% DOE cost share.

The LPMEOH™ Demonstration Unit operated at 100% availability during this quarter. There were four syngas outages experienced during the quarter on 23 April 2001 (17 hours), 08 May 2001 (13 hours), 24 May 2001 (150 hours), and 18 June 2001 (23 hours), respectively. The planned biennial LPMEOH™ Demonstration Unit outage, which began on 28 March 2001, was completed during this quarter on 20 April 2001 (472 hours). The LPMEOH™ Demonstration Unit was also shutdown for the planned biennial Eastman complex-wide outage on 13 May 2001 and restarted on 22 May 2001 (224 hours).

The scheduled biennial inspection of all pressure vessels within the LPMEOH™ Demonstration Unit, as required by Tennessee state code, was performed. Catalyst slurry

was pressure transferred from the LPMEOH™ Reactor to the slurry tank (29D-02) and held during the outage period. No issues were observed with any of the units evaluated as part of the code inspection. Maintenance activities were also completed during this outage. These activities included cleaning of all heat exchangers, cleaning all of the differential pressure measurement devices in slurry service, and repairing the boiler feedwater control valve (FV-106). The reactor syngas inlet sample point (No. 5) was moved to a location upstream of the economizer to reduce the probability of oil or slurry entering the columns of the gas chromatographs during operation. The engineering modifications required for the reduction of the adsorbent in the 29C-40 catalyst guard bed and in-situ activation of methanol synthesis catalyst in the 29C-01 LPMEOH™ Reactor were also completed during the shutdown period.

The LPMEOH™ Demonstration Unit was also shut down for the biennial Eastman complex-wide outage on 13 May 2001. During this outage, the catalyst slurry was held in the LPMEOH™ Reactor and the recycle compressor was used to maintain reactor inlet flow as part of the standby conditions. No maintenance activities were conducted within the LPMEOH™ Demonstration Unit during this outage.

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During the reporting period, quantities of the syngas stream which contains primarily carbon monoxide (Carbon Monoxide Gas, or CO Gas) became available for use in CO-rich testing. A reactor inlet ratio of hydrogen (H₂) to CO of approximately 0.6:1 was used for operations conducted between 09 May 2001 and 13 May 2001. The methanol productivity met expectations for this condition.

During most of the quarter, the flowrate of the primary syngas feed (Balanced Gas) was controlled at an average flowrate of 575 KSCFH. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235°C.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Copper crystallite size measurements have not increased in the most recent samples (an increase may be an indication of catalyst deactivation). Additional updates to the catalyst poisons data are pending further analytical results.

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. During the biennial outage, the sparger was removed from the LPMEOH™ Reactor for inspection and cleaning. In addition, all differential pressure transmitters around the sparger and reactor were calibrated, and the pressure taps were cleaned. Following the restart of the unit, the resistance coefficient was higher than the

value which was calculated following the March 1999 outage. Two scenarios have been postulated: the increase in resistance coefficient is real, and possibly related to a period of high reactor level during the restart which limited the syngas flow through the reactor; or the recalibration of the differential pressure transmitters has shifted the baseline measurement. The performance of the sparger will continue to be monitored closely for changes in performance.

During this reporting period, the adsorbent material in the 29C-40 catalyst guard bed was brought onstream. This material is chemically treated with copper oxide to enhance its arsine removal capabilities. Earlier work had identified the need to reduce the copper oxide component to copper metal prior to exposure to syngas at full header pressure. The reduction procedure, using a dilute stream of Balanced Gas in nitrogen and a protocol for increasing the adsorbent temperature with time, was begun on 17 April 2001. Temperature control within the catalyst guard bed was excellent. The reduction ended on 19 April 2001. The catalyst guard bed was then cooled, and a pressure check to full header pressure with syngas was conducted. The guard bed internal temperatures increased by about 30-40°C and then stabilized following the introduction of the syngas. This was consistent with the temperature rise which would be expected due to the adsorption of CO on the guard bed material.

The catalyst guard bed was placed in service at 1500 hrs on 26 April 2001. There were no incidents resulting from pressurizing or initial contact with the Balanced Gas feed stream. During the week of 30 April 2001, Air Products analytical personnel monitored the initial performance of the catalyst guard bed for removal of arsenic. Preliminary results indicated that breakthrough of arsine may have already occurred. Additional laboratory and field test work is ongoing to confirm these results as well as determine the mechanism for the early breakthrough of arsine through the catalyst guard bed. It is possible that damage to the adsorbent material may have occurred during the August 2000 temperature excursion when this material was first placed into service. Pending results of the additional testing, a decision to remove and replace the existing adsorbent will be made.

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During this quarter, planning, procurement, and test operations were concluded at the project sites selected for the off-site, product-use test program. Work has begun on the draft Topical Report which summarizes the objectives and results of this product-use test program. A paper from the 1999 Pittsburgh Coal Conference is being used as the basis for providing the Clean Coal prospective on the various tests and a summary of the results; the individual reports will be included in supplementary volumes to the Topical Report. A release of the draft report and supplementary volumes is targeted for September of 2001.

Activities associated with Design Verification Testing (DVT) of the Liquid Phase Dimethyl Ether (LPDME) Process have been completed. A Topical Report, which presents the results

of the DVT at the LaPorte AFDU, was approved by DOE and issued. The LPMEOH™ Demonstration Project will prepare a separate Topical Report on the market analysis for DME and review of the economics of the LPDME Process.

A DOE quarterly review meeting was held during the week of 25 June 2001 in Pittsburgh. The performance of the LPMEOH™ Demonstration Unit since the last meeting (January 2001) was the primary topic of discussion.

A draft of the paper entitled “Clean Liquid Fuels and Chemicals from Coal” was submitted to DOE for review. This paper will be presented at the U.S.-China Clean Energy Technology Forum (29 August - 01 September 2001).

The abstract for the paper entitled “Operating Overview for the Liquid Phase Methanol (LPMEOH™) Process Demonstration Unit at Kingsport, Tennessee” was approved by DOE and was accepted for presentation at the Gasification Technologies Conference (07-10 October 2001).

Air Products has been asked to participate in the poster session for the Clean Coal and Power Conference (19-20 November 2001). The abstract entitled “Liquid Phase Methanol (LPMEOH™) Process Commercial Demonstration” was approved by DOE and was accepted for presentation at this conference (formerly the Clean Coal Technology Conference).

The abstract for the paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was accepted by the conference organizers. This paper will be presented at the 18th Annual International Pittsburgh Coal Conference (03-07 December 2001).

One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2001. Seventy-five percent (75%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2001.

A. Introduction

The Liquid Phase Methanol (LPMEOH™) demonstration project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L. P. (the Partnership). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. A demonstration unit producing 80,000 gallons per day (260 TPD) of methanol was designed, constructed, and is operating at a site located at the Eastman chemicals-from-coal complex in Kingsport. The Partnership will own and operate the facility for the demonstration period.

This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to “demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility.” The project has been

demonstrating the suitability of the methanol produced for use as a chemical feedstock or as a low-sulfur dioxide, low-nitrogen oxides alternative fuel in stationary and transportation applications. The project has also evaluated the demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol.

The LPMEOH™ Process is the product of a cooperative development effort by Air Products and the DOE in a program that started in 1981. It was successfully piloted at a 10-TPD rate in the DOE-owned experimental unit at Air Products' LaPorte, Texas, site. This demonstration project is the culmination of that extensive cooperative development effort.

B. Project Description

The demonstration unit, which occupies an area of 0.6 acre, is integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex employs approximately 8,600 people. In 1983, Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas (syngas) generated by this gasification facility is used to produce carbon monoxide and methanol. Both of these products are used to produce methyl acetate and ultimately cellulose acetate and acetic acid. The availability of this highly reliable coal gasification facility was the major factor in selecting this location for the LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas or H₂ Gas, carbon monoxide gas or CO Gas, and the primary syngas feed known as Balanced Gas) are diverted from existing operations to the LPMEOH™ Demonstration Unit, thus providing the range of coal-derived syngas ratios (hydrogen to carbon monoxide) needed to meet the technical objectives of the demonstration project.

For descriptive purposes and for design and construction scheduling, the project has been divided into four major process areas with their associated equipment:

- *Reaction Area* - Syngas preparation and methanol synthesis reaction equipment.
- *Purification Area* - Product separation and purification equipment.
- *Catalyst Preparation Area* - Catalyst and slurry preparation and disposal equipment.
- *Storage/Utility Area* - Methanol product, slurry, and oil storage equipment.

The physical appearance of this facility closely resembles the adjacent Eastman process plants, including process equipment in steel structures.

- *Reaction Area*

The reaction area includes feed gas compressors, catalyst guard beds, the reactor, a steam drum, separators, heat exchangers, and pumps. The equipment is supported by a matrix of structural steel. The most salient feature is the reactor, since with supports, it is approximately 84-feet tall.

- *Purification Area*

The purification area features two distillation columns with supports; one is approximately 82-feet tall, and the other 97-feet tall. These vessels resemble the columns of the surrounding process areas. In addition to the columns, this area includes the associated reboilers, condensers, air coolers, separators, and pumps.

- *Catalyst Preparation Area*

The catalyst preparation area consists of a building with a roof and partial walls, in which the catalyst preparation vessels, slurry handling equipment, and spent slurry disposal equipment are housed. In addition, a hot oil utility system is included in the area.

- *Storage/Utility Area*

The storage/utility area includes two diked lot-tanks for methanol, two tanks for oil storage, a slurry holdup tank, a trailer loading/unloading area, and an underground oil/water separator. A vent stack for safety relief devices is located in this area.

C. Process Description

The LPMEOH™ Demonstration Unit is integrated with Eastman's coal gasification facility. A simplified process flow diagram is included in Appendix A. Syngas is introduced into the slurry reactor, which contains a slurry of liquid mineral oil with suspended solid particles of catalyst. The syngas dissolves through the mineral oil, contacts the catalyst, and reacts to form methanol. The heat of reaction is absorbed by the slurry and is removed from the slurry by steam coils. The methanol vapor leaves the reactor, is condensed to a liquid, sent to the distillation columns for removal of higher alcohols, water, and other impurities, and is then stored in the day tanks for sampling before being sent to Eastman's methanol storage. Most of the unreacted syngas is recycled back to the reactor with the syngas recycle compressor, improving cycle efficiency. The methanol will be used for downstream feedstocks and has been used in off-site, product-use testing to determine its suitability as a transportation fuel and as a fuel for stationary applications in the power industry.

D. Results and Discussion

The project status is reported by task, covering those areas in which activity took place during the reporting period. Major accomplishments during this period are as follows:

D.1 Off-Site Testing (Product-Use Demonstration)

The LPMEOH™ Demonstration Project has completed the testing of stabilized methanol from both the LaPorte AFDU and the Kingsport LPMEOH™ Demonstration Unit in various off-site mobile and stationary applications. The product-use test program was developed to enhance the early commercial acceptance of central clean coal technology processing

facilities, coproducing electricity and methanol to meet the needs of the local community. One of the advantages of the LPMEOH™ Process for coproduction from coal-derived syngas is that the as-produced, stabilized (degassed) methanol product is of unusually high quality (e.g. less than 1 wt% water) which may be suitable for the premium fuel applications. When compared to conventional methanol synthesis processes, cost savings (10 to 15%) of several cents per gallon of methanol can be achieved in coproduction facilities, if the suitability of the stabilized product can be demonstrated.

Product-use tests commenced during the first year of demonstration operations. An inventory of approximately 12,000 gallons of stabilized methanol was produced at LPMEOH™ Demonstration Unit in February of 1998 to supply the needs of the product-use test program; due to the pre-1998 timing for certain tests, methanol was shipped from the inventory produced and held at the LaPorte AFDU.

Work has begun on the draft Topical Report which summarizes the objectives and results of this product-use test program. A paper from the 1999 Pittsburgh Coal Conference is being used as the basis for providing the Clean Coal prospective on the various tests and a summary of the results; the individual reports will be included in supplementary volumes. A release of the draft report is targeted for September of 2001.

D.2 DME Design Verification Testing

The LPMEOH™ Demonstration Project has completed Design Verification Testing (DVT) to coproduce dimethyl ether (DME) with methanol via the Liquid Phase Dimethyl Ether (LPDME) Process. DVT was required to provide additional data for engineering design and evaluation of the potential for demonstration at the LPMEOH™ Demonstration Unit. The essential steps required for decision-making were: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks.

Execution of the LPDME DVT at the LaPorte AFDU was completed during October and November of 1999, and preliminary results from the operation were presented in Technical Progress Report No. 22. Results from a cost estimate for a commercial-scale LPDME plant were presented in Technical Progress Report No. 23. After discussing the results from the LPDME DVT activities and the ongoing performance results from Kingsport, the project participants agreed that the available resources should be directed toward improving the catalyst performance for the LPMEOH™ Process during the remaining time within the operating program; any improvement in the catalyst performance for the methanol synthesis catalyst will also yield benefits for the LPDME catalyst system.

A Topical Report, which presents the results of the DVT at the LaPorte AFDU, has been issued.

The LPMEOH™ Demonstration Project will prepare a separate Topical Report on the market analysis for DME and review of the economics of the LPDME Process.

D.3 LPMEOH™ Process Demonstration Unit - Methanol Operation

Table D.3-1 contains the summary table of performance data for the LPMEOH™ Demonstration Unit during the reporting period. These data represent daily averages, typically from a 24-hour material balance period, and those days with less than 12 hours of stable operation are omitted. Appendix B contains samples of the detailed material balance reports which are representative of the operation of the LPMEOH™ Demonstration Unit during the reporting period.

During the reporting period, a total of 2,655,172 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No environmental incidents or injuries were reported during this quarter.

The LPMEOH™ Demonstration Unit operated at 100% availability during this quarter. Appendix C, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter. There were four syngas outages experienced during the quarter on 23 April 2001 (17 hours), 08 May 2001 (13 hours), 24 May 2001 (150 hours), and 18 June 2001 (23 hours), respectively. The planned biennial LPMEOH™ Demonstration Unit outage, which began on 28 March 2001, was completed during this quarter on 20 April 2001 (472 hours). The LPMEOH™ Demonstration Unit was also shutdown for the planned biennial Eastman complex-wide outage on 13 May 2001 and restarted on 22 May 2001 (224 hours).

LPMEOH™ Demonstration Unit Inspection – March – April 2001

The scheduled biennial inspection of all pressure vessels within the LPMEOH™ Demonstration Unit, as required by Tennessee state code, was performed. Catalyst slurry was pressure transferred from the LPMEOH™ Reactor to the slurry tank (29D-02) and held during the outage period. No issues were observed with any of the units evaluated as part of the code inspection.

The key observations during the outage included the following:

- All cooling water heat exchangers - no signs of fouling
- Pressure Vessels - no signs of corrosion, damage to welds, or stress
- Recycle Compressor (29K-01) - evidence of abrasive wear on inlet vanes; arrangements are being made for a vendor representative to perform an inspection of the compressor
- Reactor - no signs of corrosion or erosion; the sparger was removed, inspected and cleaned, and the differential pressure transmitters were recalibrated and the taps were cleaned
- Methanol lot tanks - no signs of corrosion
- Syngas economizer (29E-02) - this heat exchanger, which condenses process oil from the reactor effluent stream, showed a layer catalyst in the tubes; hydroblasting was performed to clean the exchanger

Table D.3-1. Data Summary for LPMEOH™ Demonstration Unit

Case	Date	Days	Gas	Temp	Pres.	Fresh	Recycle	Reactor	Purge	Inlet Sup.	Space	Slurry	Gas	Gassed	Catalyst	Catalyst	CO	Reactor	Syngas	Raw	Catalyst	Reactor	U	Sparger	Sparger
		On Stream	Type	(Deg C)	(psig)	Feed (KSCFH)	Gas (KSCFH)	Feed (H ₂ -CO)	Gas (KSCFH)	Velocity (ft/sec)	Velocity (l/hr-kg)	Conc. (wt% ox)	Holdup (vol%)	Slurry Hgt (ft)	Inventory (lb)	Age (eta)	Conv. (%)	O-T-M Conv. (%)	Util. (SCF/lb)	MeOH Prod. (TPD)	MeOH Prod. (gmol/hr-kg)	Vol. Prod. (TPD/ft ³)	Overall (ft ² F)	dP (psi)	Resist. ("K")
11	29-Apr-01	1226	Balanced	230	710	543	2,344	2.94	40.2	0.65	3577	43.4	29.2	51.5	46,716	0.249	24.5	16.2	40.3	161.7	9.03	0.075	108	8.2	7.96
11	4-May-01	1231	Balanced	235	710	621	2,258	2.88	65.6	0.66	3584	43.4	27.4	50.5	46,716	0.245	24.9	17.8	41.5	179.7	10.04	0.085	102	8.14	7.43
5	11-May-01	1238	0.6:1	235	710	400	2,486	0.68	37.9	0.67	3634	43.7	24.4	48	46,716	0.222	7.9	11.4	41.6	115.1	6.44	0.057	112	12.88	6.6
5	12-May-01	1239	0.6:1	233	685	462	2,334	0.61	75.7	0.66	3484	44.5	27.6	48.5	46,716	0.259	7.9	12	46.9	118	6.61	0.058	142	12.64	6.74
11	31-May-01	1258	Balanced	235	710	544	2,224	2.73	74.7	0.63	3420	43.4	21.1	46.5	46,716	0.198	22.0	15.3	44	148.5	8.31	0.076	105	9.3	9.55
11	3-Jun-01	1261	Balanced	234	710	603	2,063	2.70	115.8	0.61	3302	44.3	28.4	49.5	46,716	0.212	23.4	16.4	46.7	154.8	8.68	0.075	106	6.05	6.78
11	5-Jun-01	1263	Balanced	233	710	571	2,192	2.36	138.8	0.62	3859	39.5	14.4	44	41,216	0.222	18.1	14	49.4	138.6	8.82	0.075	107	5.62	5.89
11	6-Jun-01	1264	Balanced	234	710	572	2,178	2.30	131	0.62	3847	43.1	19.9	41	41,216	0.222	18.4	14.4	48.1	142.6	9.07	0.083	126	6.18	6.34
11	7-Jun-01	1265	Balanced	234	710	574	2,151	2.54	99	0.62	3680	45.5	25.9	42	42,866	0.238	21.2	15.6	45.4	151.6	9.3	0.086	124	6.39	6.93
11	8-Jun-01	1266	Balanced	234	710	612	2,110	2.50	46.4	0.62	3474	42.1	21.5	48	45,620	0.266	25.9	19	40.7	180.7	10.35	0.09	105	7.7	7.48
11	14-Jun-01	1272	Balanced	235	710	671	2,010	3.24	105.6	0.61	3372	41.2	22	50	45,620	0.276	31.4	19.2	44.7	179.9	10.33	0.086	118	6.19	7.37
11	15-Jun-01	1273	Balanced	235	710	608	2,127	2.60	68.7	0.63	3500	42.7	25.7	49.5	45,620	0.242	25.0	17.7	42.7	170.8	9.79	0.082	99	7.02	6.74
11	16-Jun-01	1274	Balanced	235	710	612	2,097	2.92	87.8	0.62	3446	43	25.1	48.5	45,620	0.237	26.5	17.5	44.1	166.5	9.55	0.082	103	6.77	7.26
11	17-Jun-01	1275	Balanced	235	710	614	2,116	2.93	95.4	0.62	3467	42.6	24.7	49.0	45,620	0.232	26.0	17.1	44.9	164.2	9.42	0.08	99	6.56	7.00
11	19-Jun-01	1277	Balanced	235	710	556	2,170	2.90	72.6	0.62	3462	44.6	32.4	50.5	45,620	0.219	24.0	15.8	43.4	153.5	8.8	0.072	104	6.43	6.98
11	20-Jun-01	1278	Balanced	235	710	583	2,121	3.19	83.4	0.61	3411	39.8	22.2	53.0	45,620	0.237	27.1	16.7	43.8	159.7	9.16	0.072	104	5.98	7.04
11	21-Jun-01	1279	Balanced	235	710	577	2,154	2.98	76.1	0.62	3468	39.7	19.6	51.5	45,620	0.233	25.6	16.5	43.3	159.8	9.16	0.074	105	5.84	6.30
11	22-Jun-01	1280	Balanced	235	710	584	2,161	2.78	68.9	0.63	3506	40.9	23.3	51.5	45,620	0.236	24.8	16.9	42.7	164.1	9.4	0.076	109	6.6	6.67
11	23-Jun-01	1281	Balanced	235	710	581	2,163	2.91	78.6	0.63	3478	40.6	22.5	51.5	45,620	0.233	25.0	16.5	43.6	159.9	9.16	0.074	103	5.99	6.44
11	24-Jun-01	1282	Balanced	235	710	579	2,210	2.76	78.4	0.63	3512	40.6	24.8	53.0	45,620	0.227	23.8	16.3	43.5	159.8	9.16	0.072	97	6.01	6.21
11	25-Jun-01	1283	Balanced	235	710	580	2,221	2.63	72.5	0.64	3551	40.9	22.7	51.0	45,620	0.227	23.2	16.5	42.8	162.7	9.32	0.076	103	6.67	6.59
11	26-Jun-01	1284	Balanced	235	710	592	2,175	2.61	73.1	0.63	3519	40.4	18.7	49.5	45,620	0.233	23.8	16.9	42.7	166.2	9.52	0.08	110	6.11	6.08
11	28-Jun-01	1286	Balanced	235	710	568	2,196	2.69	66.6	0.63	3500	43.4	20.2	45.0	45,620	0.227	23.8	16.5	42.5	160.4	9.19	0.085	114	6.41	6.43
11	30-Jun-01	1288	Balanced	235	710	573	2,160	2.58	62.5	0.63	3477	42.5	23.5	48.5	45,620	0.237	23.6	16.8	42.2	162.9	9.33	0.080	119	6.54	6.53

Maintenance activities were also performed during this outage. These activities included cleaning of all heat exchangers, cleaning all of the differential pressure measurement devices in slurry service, and repairing the boiler feedwater control valve (FV-106). The reactor syngas inlet sample point (No. 5) was moved to a location upstream of the economizer to reduce the probability of oil or slurry entering the columns of the gas chromatographs during operation. The engineering modifications required for the reduction of the adsorbent in the 29C-40 catalyst guard bed and in-situ activation of methanol synthesis catalyst in the 29C-01 LPMEOH™ Reactor were also completed during the shutdown period.

The LPMEOH™ Demonstration Unit was also shut down for the biennial Eastman complex-wide outage on 13 May 2001. During this outage, the catalyst slurry was held in the LPMEOH™ Reactor and the recycle compressor was used to maintain reactor inlet flow as part of the standby conditions. No maintenance activities were conducted within the LPMEOH™ Demonstration Unit during this outage.

Catalyst Life (η) – April - June 2001

The “age” of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable η , which is defined as the ratio of the rate constant at any time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave). Appendix C, Figure 1 plots $\log \eta$ versus days onstream from September of 2000 to the end of the reporting period. Since catalyst activity typically follows a pattern of exponential decay, the plot of $\log \eta$ is fit to a series of straight lines, with step-changes whenever fresh catalyst was added to the reactor.

A catalyst withdrawal and addition campaign was undertaken during the quarter to raise catalyst activity. A series of two withdrawals were conducted on 04 and 05 June 2001. Two batches of fresh catalyst were activated and added to the reactor on 07 and 08 June 2001, respectively. After the second batch of freshly activated catalyst was transferred into the reactor, the catalyst inventory was calculated to be 45,620 pounds. There were no operating periods of sufficient duration (minimum of about 2 weeks) to calculate a catalyst deactivation rate.

During the reporting period, additional CO Gas became available for use in CO-rich testing. A reactor inlet $H_2:CO$ ratio of approximately 0.6:1 was used for operations conducted between 09 May 2001 and 13 May 2001. The methanol productivity met expectations for this condition.

During most of the quarter, the flowrate of Balanced Gas was controlled at an average flowrate of 575 KSCFH. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235°C.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Appendix C, Table 2 summarizes the results to date. Copper crystallite size measurements have not increased in the most recent samples (an increase may be an indication of catalyst deactivation). Additional updates to the catalyst poisons data are pending further analytical results.

Sparger Resistance

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. Appendix C, Figure 2 plots the average daily sparger resistance coefficient for the reporting period. The data for this plot, along with the corresponding average pressure drop, are also included in Table D.3-1.

During the biennial outage, the sparger was removed from the LPMEOH™ Reactor for inspection and cleaning. In addition, all differential pressure transmitters around the sparger and reactor were calibrated, and the pressure taps were cleaned. Following the restart of the unit, the resistance coefficient was higher than the value which was calculated following the March 1999 outage. Two scenarios have been postulated: the increase in resistance coefficient is real, and possibly related to a period of high reactor level during the restart which limited the syngas flow through the reactor; or the recalibration of the differential pressure transmitters has shifted the baseline measurement. The performance of the sparger will continue to be monitored closely for changes in performance.

29C-40 Catalyst Guard Bed Adsorbent Reduction and Initial Operation

In August 2000, the manganese oxide adsorbent in the 29C-40 catalyst guard bed was replaced with a commercially available activated carbon adsorbent. This adsorbent material is chemically treated with copper oxide to enhance its arsine removal capabilities. The selection of this adsorbent material followed extensive investigative work in the laboratory and at Kingsport to evaluate the effectiveness of the material and to address questions on potential interaction with the components of the syngas feed. Upon the initial exposure of the adsorbent material to Balanced Gas, a temperature excursion was experienced. Follow-up work indicated that the copper oxide component of the adsorbent needed to be reduced in a controlled manner prior to contact with the Balanced Gas feed stream. Modifications to the piping and instrumentation systems were performed during the biennial outage (March 2001) so that the adsorbent in the catalyst guard bed could be reduced with a dilute stream of Balanced Gas in nitrogen in a temperature controlled manner.

The reduction procedure for the adsorbent material in the catalyst guard bed was begun on 17 April 2001. Temperature control within the catalyst guard bed was excellent. The reduction ended on 19 April 2001. The catalyst guard bed was then cooled, and a pressure check to full header pressure with syngas was conducted. The guard bed internal temperatures increased by about 30-40°C and then stabilized following the introduction of the syngas. This was consistent with the temperature rise which would be expected due to the adsorption of CO on the guard bed material.

The catalyst guard bed was placed in service at 1500 hrs on 26 April 2001. There were no incidents resulting from pressurizing or initial contact with the Balanced Gas feed stream. During the week of 30 April 2001, Air Products analytical personnel monitored the initial performance of the catalyst guard bed. The technique involved sampling a slip stream of syngas from either the guard bed inlet or outlet streams, passing this slip stream across a laboratory-scale adsorbent bed, and analyzing this material for arsenic. Preliminary results

indicated that breakthrough of arsine may have already occurred. Additional laboratory and field test work is ongoing to confirm these results as well as determine the mechanism for the early breakthrough of arsine through the catalyst guard bed. It is possible that damage to the adsorbent material may have occurred during the August 2000 temperature excursion when this material was first placed into service. Pending results of the additional testing, a decision to remove and replace the existing adsorbent will be made.

D.4 Planning and Administration

A quarterly review meeting was held with DOE during the week of 25 June 2001 in Pittsburgh. The performance of the LPMEOH™ Demonstration Unit since the last meeting (January 2001) was the primary topic of discussion. The agenda, extracts from the handouts, and the notes for each meeting are included in Appendix D.

A draft of the paper entitled “Clean Liquid Fuels and Chemicals from Coal” was submitted to DOE for review. This paper will be presented at the U.S.-China Clean Energy Technology Forum (29 August - 01 September 2001).

The abstract for the paper entitled “Operating Overview for the Liquid Phase Methanol (LPMEOH™) Process Demonstration Unit at Kingsport, Tennessee” was approved by DOE and was accepted for presentation at the Gasification Technologies Conference (07-10 October 2001).

Air Products has been asked to participate in the poster session for the Clean Coal and Power Conference (19-20 November 2001). The abstract entitled “Liquid Phase Methanol (LPMEOH™) Process Commercial Demonstration” was approved by DOE and was accepted for presentation at this conference (formerly the Clean Coal Technology Conference).

A draft abstract for the paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was accepted by the conference organizers. This paper will be presented at the 18th Annual International Pittsburgh Coal Conference (03-07 December 2001).

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 June 2001, are included in Appendix E. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure (WBS) tasks. One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2001. Seventy-five percent (75%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2001.

The monthly reports for April, May, and June were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report.

E. Planned Activities for the Next Quarter

- Continue to analyze catalyst slurry samples and reactor performance data to determine causes for deactivation of methanol synthesis catalyst.
- Perform the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor.
- After the completion of the in-situ activation, continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan. Focus activities on temperature programming to maintain the required methanol productivity, monitoring catalyst activity, and monitoring the performance of the gas sparger in the reactor.
- Issue a draft Topical Report on the objectives and results of the off-site, product-use test program for stabilized methanol from the LPMEOH™ Process.
- Schedule a Project Review Meeting with DOE.

F. Conclusion

The LPMEOH™ Demonstration Unit operated at 100% availability during this quarter. There were four syngas outages experienced during the quarter on 23 April 2001 (17 hours), 08 May 2001 (13 hours), 24 May 2001 (150 hours), and 18 June 2001 (23 hours), respectively. The planned biennial LPMEOH™ Demonstration Unit outage, which began on 28 March 2001, was completed during this quarter on 20 April 2001 (472 hours). The LPMEOH™ Demonstration Unit was also shutdown for the planned biennial Eastman complex-wide outage on 13 May 2001 and restarted on 22 May 2001 (224 hours).

The scheduled biennial inspection of all pressure vessels within the LPMEOH™ Demonstration Unit, as required by Tennessee state code, was performed. Catalyst slurry was pressure transferred from the LPMEOH™ Reactor to the slurry tank (29D-02) and held during the outage period. No issues were observed with any of the units evaluated as part of the code inspection. Maintenance activities were also performed during this outage. These activities included cleaning of all heat exchangers, cleaning all of the differential pressure measurement devices in slurry service, and repairing the boiler feedwater control valve (FV-106). The reactor syngas inlet sample point (No. 5) was moved to a location upstream of the economizer to reduce the probability of oil or slurry entering the columns of the gas chromatographs during operation. The engineering modifications required for the reduction of the adsorbent in the 29C-40 catalyst guard bed and in-situ activation of methanol synthesis catalyst in the 29C-01 LPMEOH™ Reactor were also completed during the shutdown period.

The LPMEOH™ Demonstration Unit was also shut down for the biennial Eastman complex-wide outage on 13 May 2001. During this outage, the catalyst slurry was held in the LPMEOH™ Reactor and the recycle compressor was used to maintain reactor inlet flow

as part of the standby conditions. No maintenance activities were conducted within the LPMEOH™ Demonstration Unit during this outage.

A catalyst withdrawal and addition campaign was undertaken during the quarter to raise catalyst activity. A series of two withdrawals were conducted on 04 and 05 June 2001. Two batches of fresh catalyst were activated and added to the reactor on 07 and 08 June 2001, respectively. After the second batch of freshly activated catalyst was transferred into the reactor, the catalyst inventory was calculated to be 45,620 pounds. There were no operating periods of sufficient duration (minimum of about 2 weeks) to calculate a catalyst deactivation rate.

During the reporting period, additional CO Gas became available for use in CO-rich testing. A reactor inlet H₂:CO ratio of approximately 0.6:1 was used for operations conducted between 09 May 2001 and 13 May 2001. The methanol productivity met expectations for this condition.

During most of the quarter, the flowrate of Balanced Gas was controlled at an average flowrate of 575 KSCFH. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235°C.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Copper crystallite size measurements have not increased in the most recent samples (an increase may be an indication of catalyst deactivation). Additional updates to the catalyst poisons data are pending further analytical results.

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. During the biennial outage, the sparger was removed from the LPMEOH™ Reactor for inspection and cleaning. In addition, all differential pressure transmitters around the sparger and reactor were calibrated, and the pressure taps were cleaned. Following the restart of the unit, the resistance coefficient was higher than the value which was calculated following the March 1999 outage. Two scenarios have been postulated: the increase in resistance coefficient is real, and possibly related to a period of high reactor level during the restart which limited the syngas flow through the reactor; or the recalibration of the differential pressure transmitters has shifted the baseline measurement. The performance of the sparger will continue to be monitored closely for changes in performance.

During this reporting period, the adsorbent material in the 29C-40 catalyst guard bed was brought onstream. This material is chemically treated with copper oxide to enhance its arsine removal capabilities. Earlier work had identified the need to reduce the copper oxide component to copper metal prior to exposure to syngas at full header pressure. The reduction procedure, using a dilute stream of Balanced Gas in nitrogen and a protocol for increasing the adsorbent temperature with time, was begun on 17 April 2001. Temperature control within the catalyst guard bed was excellent. The reduction ended on 19 April 2001. The catalyst guard bed was then cooled, and a pressure check to full header pressure with syngas was conducted. The guard bed internal temperatures increased by about 30-40°C and

then stabilized following the introduction of the syngas. This was consistent with the temperature rise which would be expected due to the adsorption of CO on the guard bed material.

The catalyst guard bed was placed in service at 1500 hrs on 26 April 2001. There were no incidents resulting from pressurizing or initial contact with the Balanced Gas feed stream. During the week of 30 April 2001, Air Products analytical personnel monitored the initial performance of the catalyst guard bed for removal of arsenic. Preliminary results indicated that breakthrough of arsine may have already occurred. Additional laboratory and field test work is ongoing to confirm these results as well as determine the mechanism for the early breakthrough of arsine through the catalyst guard bed. It is possible that damage to the adsorbent material may have occurred during the August 2000 temperature excursion when this material was first placed into service. Pending results of the additional testing, a decision to remove and replace the existing adsorbent will be made.

During the reporting period, a total of 2,655,172 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 79.1 million gallons of methanol have been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations were concluded at the project sites selected for the off-site, product-use test program. Work has begun on the draft Topical Report which summarizes the objectives and results of this product-use test program. A paper from the 1999 Pittsburgh Coal Conference is being used as the basis for providing the Clean Coal prospective on the various tests and a summary of the results; the individual reports will be included in supplementary volumes as noted previously. A release of the draft report and supplementary volumes is targeted for September of 2001.

Activities associated with Design Verification Testing (DVT) of the LPDME Process have been completed. A Topical Report, which presents the results of the DVT at the LaPorte AFDU, was approved by DOE and issued. The LPMEOH™ Demonstration Project will prepare a separate Topical Report on the market analysis for DME and review of the economics of the LPDME Process.

A DOE quarterly review meeting was held during the week of 25 June 2001 in Pittsburgh. The performance of the LPMEOH™ Demonstration Unit since the last meeting (January 2001) was the primary topic of discussion.

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The abstract for the paper entitled "Operating Overview for the Liquid Phase Methanol (LPMEOH™) Process Demonstration Unit at Kingsport, Tennessee" was approved by DOE and was accepted for presentation at the Gasification Technologies Conference (07-10 October 2001).

Air Products has been asked to participate in the poster session for the Clean Coal and Power Conference (19-20 November 2001). The abstract entitled "Liquid Phase Methanol (LPMEOH™) Process Commercial Demonstration" was approved by DOE and was accepted for presentation at this conference (formerly the Clean Coal Technology Conference).

A draft abstract for the paper entitled "Liquid Phase Methanol (LPMEOH™) Process Development" was accepted by the conference organizers. This paper will be presented at the 18th Annual International Pittsburgh Coal Conference (03-07 December 2001).

One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2001. Seventy-five percent (75%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2001.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

APPENDIX B - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX C - RESULTS OF DEMONSTRATION UNIT OPERATION

**Table 1 - Summary of LPMEOHTM Demonstration Unit Outages -
April/June 2001**

Table 2 - Summary of Catalyst Samples - Second Catalyst Batch

Figure 1 - Catalyst Age (η): September 2000 - June 2001

Figure 2 - Sparger Resistance Coefficient vs. Days Onstream

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - April/June 2001

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
4/1/01 00:00	4/1/01 00:00	0.0	471.7	Scheduled Biennial Outage
4/20/01 15:44	4/23/01 08:20	64.6	17.2	Syngas Outage
4/24/01 01:29	5/8/01 21:31	356.0	12.6	Syngas Outage
5/9/01 10:05	5/13/01 04:30	90.4	223.7	Scheduled Complex Shutdown
5/22/01 12:11	5/24/01 10:27	46.3	149.5	Syngas Outage
5/30/01 16:00	6/18/01 12:10	452.2	22.5	Syngas Outage
6/19/01 10:38	6/30/01 23:59	277.3		End of Reporting Period
Total Operating Hours			1286.8	
Syngas Available Hours			1286.8	
Plant Availability, %			100.00	

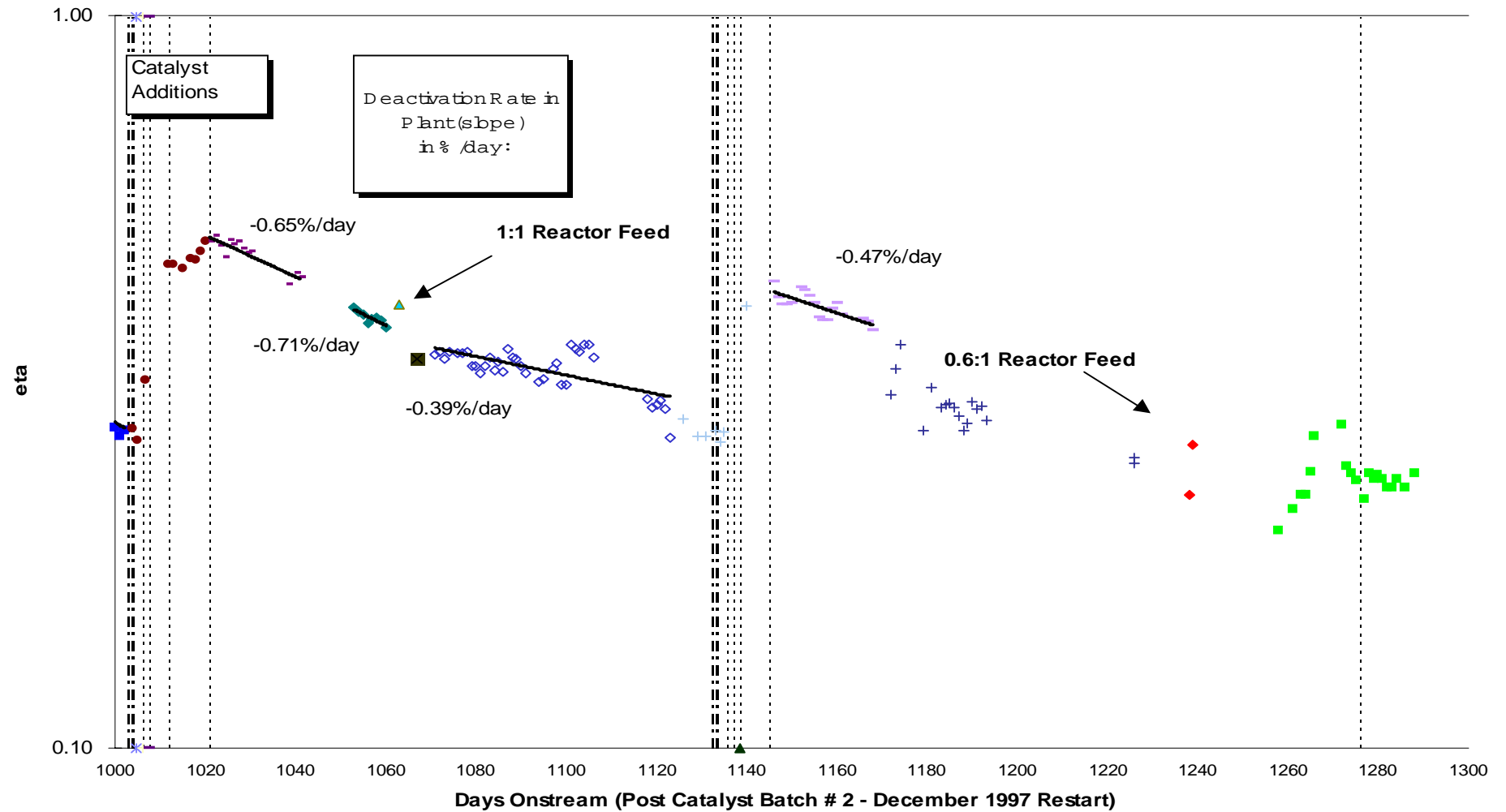
Table 2
Summary of Catalyst Samples - Second Catalyst Batch

Sample	Identity	XRD		BET	Analytical (ppmw)				
		Cu	ZnO	m ² /g	Fe	Ni	S	As	Cl
K9804-1	Reduction Sample 4/2/98 - Alternative Catalyst	72.5	84.9	105	23	11	<=110	<=12	
K9712-1	Transfer sample from 29D-02 to Reactor	95.3	74		362	47.2	66.7	10.2	nd
K9712-2	Reactor Sample Day 1	100	123.8	75	92.1	<=18	<=167	<50	nd
K9712-3	Reactor Sample Day 4	130.9	64						
K9712-4	Reactor Sample Day 10	126.8	73.3	73	126	<=22	<=127	<50	nd
K9801-2	Reactor Sample 1/26/98	132.05	98.3		63.5	39.5	42.7	29.2	<100
K9802-1	Reactor Sample 2/3/98	141.1	91.5						
K9802-2	Reactor Sample 2/9/98	158.1	113						
K9802-3	Reactor Sample 2/15/98	145.7	91		67.1	36	<=97	209	
K9802-4	Reactor Sample 2/23/98	176.8	114.5						
K9803-2	Reactor Sample 3/10/1998	154.3	95.8	44	61.4	35.8	<=94	408	
K9803-4	Reactor Sample 3/29/98	169.6	87.9						
K9804-2	Reactor Sample 4/14/98	152.4	89.3		81.7	30.8	<=170	615	
K9805-2	Reactor Sample 5/11/98	219.2	109.6		73.15	35.85	163	538	
K9806-2	Reactor Sample 6/16/98	272.3	117.2		86.4	31.1	220	1110	
K9807-2	Reactor Sample 7/8/98	263.2	108.6		88.7	27.6	277	1045	
K9807-3	Reactor Sample 7/29/98	412*	112		93.25	30.95	209	1620	
K9807-4	Reactor Sample 8/14/98	353.9*	124		121.5	37.1	213.5	1215	
K9809-1	Reactor Sample 9/24/98	347.4	129.8		69.6	29.8	326	1149	
K9810-1	Reactor Sample 10/5/98	331.1	130.4						
K9811-2	Reactor Sample 11/25/98	293.9			57.3	23.4	264	1400	<100
K9812-1	Reactor Sample 12/29/98	283.1			72.3	20.4	260	1300	<100
K9901-1	Reactor Sample 1/15/99	252.5	61.4						
K9902-1	Reactor Sample 2/17/99	474.7	133.6		82.6	22.2	385	1490	<300
K9904-3	Reactor Sample 4/27/99	417.8	110.4	15	131	18.2	348	1460	<30
K9906-1	Reactor Sample 6/1/99	517	105	43	109	19.7	316	1680	40
K9907-1	Reactor Sample 7/13/99	446	116	59	175	19.7	488	1810	30
K9908-2	Reactor Sample 8/31/99	632	117	56	161	15.1	406	1470	50
K9909-2	Reactor Sample 9/21/99	357	109	64	132	11.2	253	1050	nd
K9910-2	Reactor Sample 10/19/99	135	94	55	157	15.4	343	1270	30
K9911-1	Reactor Sample 11/4/99				184	12.8	335	1580	na
K9912-1	Reactor Sample 12/8/99	797	121	60	167	13.9	248	1400	40
K0001-1	Reactor Sample 1/5/00	613	105	63	199	10.8	292	1190	nd
K0001-2	Reactor Sample 1/19/00				205	10.0	432	1250	na
K0003-1	Reactor Sample 3/2/00	187	88.7	67	137	8.2	226	1010	30
K004-1	Reactor Sample 4/23/00	175	114.5	59	164	6.6	248	1240	20
K0007-1	Reactor Sample 7/18/00	174	107.5	69	166	< 9.6	349	1270	30
K0008-1	Reactor Sample 8/31/00	385	90.4	66	186	< 10	379	1080	50
K0009-3	Reactor Sample 9/19/00	157	96	67	145	< 10	273	1390	nd
K0011-1	Reactor Sample 11/7/00	248	79.6	70	120	< 10	237	1490	nd
K0011-2	Reactor Sample 11/27/00	263	109.2		128	< 10	258	1470	20
K0012-2	Reactor Sample 12/19/01				100	< 10	410	1480	
K0101-1	Reactor Sample 1/3/01	280	110	66	166	< 10	355	1980	30
K0102-1	Reactor Sample 2/7/01	172	104	73	121	< 10	375	1750	40
	Reactor Sample 3/7/01	164	138	69	90	< 10	416	1830	20
K0103-1	Reactor Sample 6/5/01	243	103	62					

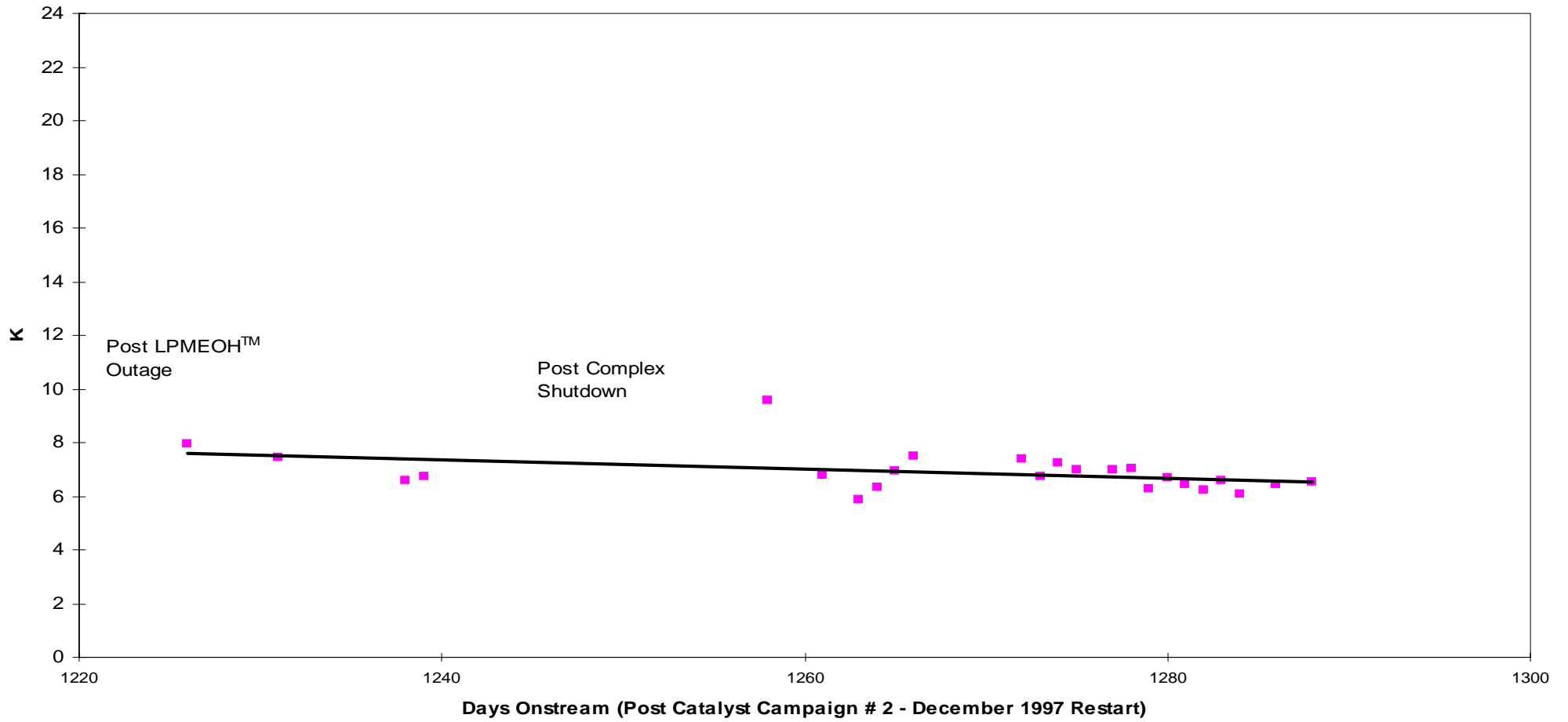
Notes:

- 1) nd = none detected
- 3) na = data not available

**Figure 1 - Kingsport LPMEOH™ Catalyst Age (eta):
September 2000 - June 2001**



**Figure 2 - Kingsport LPMEOH™
Sparger Resistance Coefficient**



APPENDIX D - PROJECT REVIEW MEETING (26 JUNE 2001)

**APPENDIX E - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT
REPORTS**